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ELECTROCHEMICAL CYCLIZATION OF UNSATURATED HYDROXY COMPOUNDS. PART II. PHENYLSELENOLACTONIZATION

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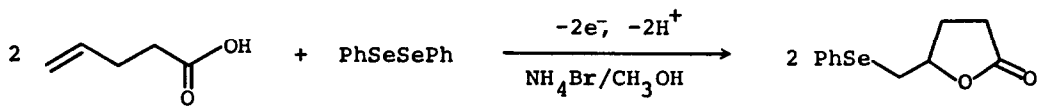
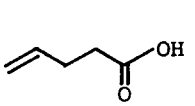
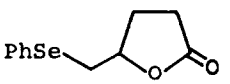
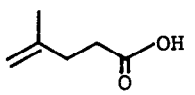
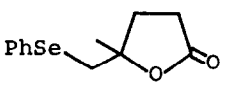
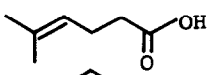
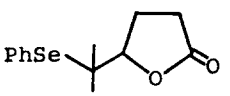
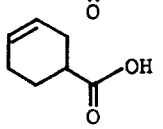
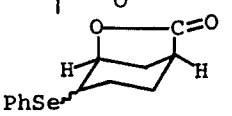
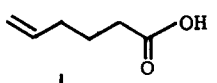
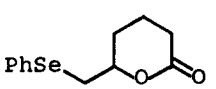
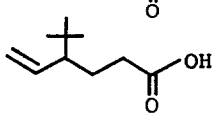
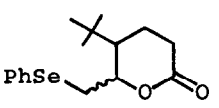
Abstract: Phenylselenolactonization was performed, in one step, by electrolysis of Δ^4- and Δ^5-unsaturated carboxylic acids and diphenyl diselenide in methanol containing ammonium bromide.

Phenylselenolactonization is known as a means of functionalizing olefinic carboxylic acids, which undergo cyclization when treated with areneselenenyl halides[1,2]. We wish to report here our studies on the extension of this process to electrochemical selenocyclofunctionalization[3]. When unsaturated carboxylic acids (such as 4-pentenoic acid) and diphenyl diselenide were electrolyzed in methanol solution of ammonium bromide (as electrolyte), phenylselenolactones were obtained in good to very good yield (56-78%) (Table). The electrolysis was performed in an undivided cell, placed in an ice-acetone-salt bath, using a grafite stick as anode and Cu foil as catode[4]. For 1 mmol of substrate, 0.5 mmol of PhSeSePh, 300 mg of NH_4Br and 5 ml of MeOH were used, the constant current being 400 mA (6 F/mol). After completion, MeOH was distilled off, the residue extracted with diethyl ether, and the crude product (upon solvent removal) purified by column chromatography (SiO_2, CH_2Cl_2).



The results in the Table show that acyclic, terminally unsubstituted and disubstituted 4-enoic acids, as well as the corresponding 3-cyclohexene-1-carboxylic acid, give only γ-lactones, whereas terminally unsubstituted 5-enoic acids afford exclusively the respective δ-lactones. 3-Butenoic and 6-heptenoic acid did not undergo intramolecular cyclization under these conditions.

Table. Electrochemical cyclization of unsaturated carboxylic acids

Unsaturated acid	Products[a]	Yield, %[b]
		71
		69
		62
		78[c]
		68
		56[c]

[a] Identified by IR and NMR spectroscopy. [b] Isolated yields.
[c] Stereochemistry, as yet, not investigated.

REFERENCES AND NOTES

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3. So far, only one such case of electrocyclization has been reported: S. Torii, K. Uneyama, M. Ono, T. Bannou, *J. Am. Chem. Soc.*, 103, 4606 (1981).
4. M. Lj. Mihailović, S. Konstantinović, R. Vukićević, *Tetrahedron Lett.*, 28, 4343 (1987).

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